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CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF CASE SCHOOL OF APPLIED SCIENCE. — XLII.

AN APPARATUS FOR CONTINUOUS VACUUM DISTILLATION.

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The occasional contributions to methods for vacuum distillation seem to indicate that a method is still wanting that shall combine convenience and efficiency. It is quite true that the various attachments that have been suggested and that are described in dealers' catalogues fall short of efficiency in essential details. In the great amount of vacuum distillation carried on in this laboratory during the last fifteen years, probably much exceeding what has been done elsewhere in a single line of work, a durable apparatus has been gradually evolved in which this work can be carried on as expeditiously as distillations under ordinary pressures.

One of the most essential features is a regulator to maintain a constant tension, and the stopcock G with lever attachment formerly described † and constantly in use is very satisfactory.

The chief features to be provided for in a convenient apparatus are the following:

1. Exclusion of air from hot oil in still during change of receiver.
2. Admission of distillates into still without interruption.
3. Admission of air into receiver before removal of each fraction.
4. Exhaustion of receiver for new fraction without connection with still.

The complete apparatus in the form used at present is shown in the following figure: —

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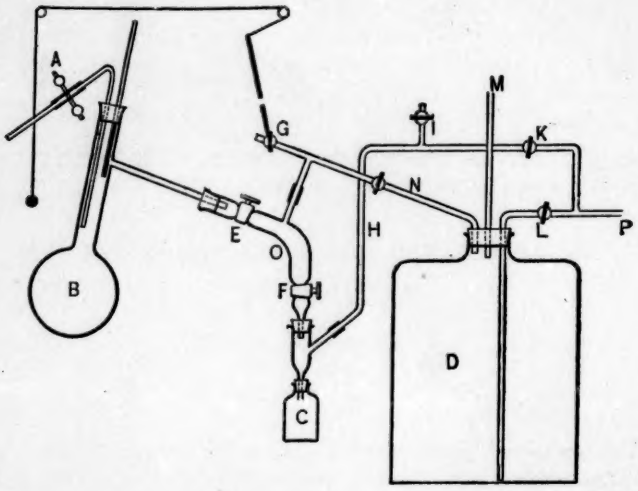
* This method is a part of the work that is carried on in this laboratory with aid granted by the Academy from the C. M. Warren fund for chemical research.

† These Proceedings, XXXI. p. 10.



The fractions are drawn into the still through the tube closed by the nipper tap A. The still is exhausted by the tube N connecting the tube O with the vacuum reservoir D. The reservoir C is exhausted by the tube H which connects with the water pump through the tube P. Air is let into the receiver C by means of the cock I, and kept from the still by the cocks E and F. The tube with the stopcocks E and F afford a convenient means for separating fractions without interruption, and without admission of air into the still. The tube M leads to the manometer. By means of a single efficient water pump the entire apparatus may be kept under a tension of 12mm. or less during continuous distillation. By means of common corks, the apparatus is readily set up and easily kept tight by the use of rubber lute.

The tubes H and N may be given less rigidity by putting them together in sections with connectors. Any water that may occasionally run back from the pump is readily drawn out if the pipe P extends to the bottom of the reservoir D.

The apparatus in this form is especially adapted for the separation of fractions with high boiling points. For very high temperatures the still must be packed in asbestos. For more volatile distillates a condenser should be inserted between the still and tube O, best by passing the exit tube of the still through the condenser. Our distillation flasks are made with a high exit tube to give a long neck, which is filled with

broken glass resting on a piece of glass rod with a head as previously described. As is evident from the figure, this apparatus may readily be set up from supplies always at hand in the laboratory, except the tube O, which any glass-blower can make.

Suggestions as to details have been made by various assistants, especially by Mr. O. J. Sieplein, instructor in chemistry, who prepared the drawing.